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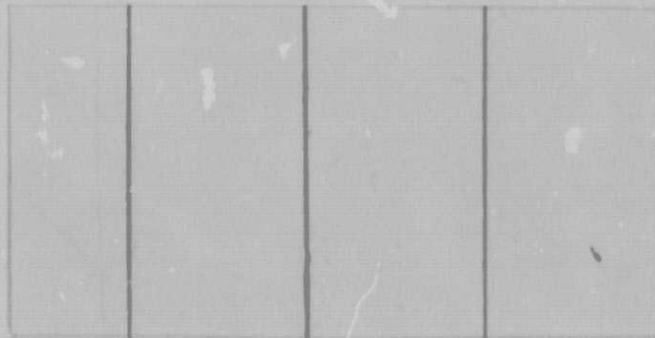


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HUNTSVILLE RESEARCH & ENGINEERING CENTER

Cummings Research Park
4800 Bradford Drive,
Huntsville, Alabama

ASTP SCIENCE DEMONSTRATION
DATA ANALYSIS

FINAL REPORT

October 1977

Contract NAS8-32222

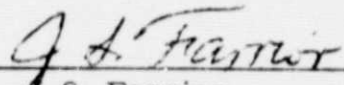
Prepared for National Aeronautics and Space Administration
Marshall Space Flight Center, Alabama 35812

by

Philomena G. Grodzka

Sidney V. Bourgeois

APPROVED: _____


J. S. Farrior
Resident Director

FOREWORD

This document is the final report for Contract NAS8-32222, performed for the National Aeronautics and Space Administration, Marshall Space Flight Center, by personnel of Lockheed Missiles & Space Company, Huntsville Research & Engineering Center, Huntsville, Alabama. Barbara Facemire of NASA-MSFC was the contract monitor for this study.

ACKNOWLEDGMENTS

The authors are grateful to Astronaut Donald K. Slayton for performing the subject demonstrations during the Apollo-Soyuz mission. Our thanks also go to Barbara Facemire who was a Co-Investigator on the demonstration and who performed the surface tension and contact angle measurements and obtained the ground based motion pictures of various phenomena.

The authors are also grateful to Dr. Robert S. Synder and Mr. K. Stuart Clifton of NASA-MSFC for their help and advice during the preparation of the experiments for flight. Drs E. Stuhlinger, A.M. Schwartz, A.C. Zettlemoyer, R.J. Good, W. Neumann, S. Ross, F.M. Fowkes, J.W. Vanderhoff, H. Leidheiser, Jr., and F.J. Micale are also to be commended for their interest, encouragement, and advice.

ABSTRACT

Analyses of the Apollo-Soyuz Science Demonstrations entitled "Chemical Foams" and "Liquid Spreading" are presented. The Chemical Foams Demonstration showed that aqueous foams and gas/liquid dispersions are more stable in low-g than on the ground. Unique chemical reactions in low-g foams and gas/liquid dispersions should therefore be possible. Further ground tests on the formaldehyde clock reaction led to the rather surprising conclusions that

- Surfaces can exert a nucleation effect.
- Long-range surface influences on chemical reaction rates are apparently operative.

These conclusions are, it is believed, original discoveries which will be reported in the open literature.

The Liquid Spreading Demonstration showed the spreading of oil on a plastic and metal surface in low-g. The low-g spreading rates fell within the bounds of existing theories, but were not precise enough to allow a decision as to which theory was the most representative.

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INTRODUCTION AND SUMMARY

The "Chemical Foams," and "Liquid Spreading" science demonstration experiments were performed on the Apollo-Soyuz mission on 19 July 1975 by Astronaut Donald K. Slayton. Motion pictures were taken of each demonstration and the film returned for analysis of space effects on the rate of chemical reaction and the rate and degree of wetting and spreading.

One part of the Chemical Foams demonstration consisted of foaming, by shaking, aqueous solutions of bromthymol blue of pH of about 2.8. The stabilities of the low-g foams were compared to those produced similarly on the ground. As expected the low-g foams were much more stable than the one-g foams.

In the other part of the Chemical Foams demonstration, a clock reaction (formaldehyde, sodium metabisulfite, sodium sulfite, and phenolphthalein as an indicator) was performed. The objective of the experiment was to compare the low-g and ground times for reaction completion. In low-g the reaction was expected to occur in stable foams or gas/liquid dispersions. It was further expected that because of Gibbean adsorption reactive species would be concentrated at liquid/gas surfaces. The reaction, therefore, was expected to occur more rapidly in low-g than on the ground. It was therefore a surprise to see on the flight film that the low-g reactions initiated at the meniscus/wall interfaces and not in the stable gas/liquid dispersions. The reaction, furthermore, exhibited in further ground tests many other facets hitherto unobserved. These ground tests along with unexpected experimental difficulties in the flight test made the experimental error difficult to assess. An unequivocal statement on whether or not the flight sample reactions actually proceeded faster than similar ground reactions, therefore, is not possible at

this time. The further ground tests, however, led to the rather surprising conclusions that:

- Surfaces can exert a nucleation effect on the formaldehyde clock reaction.
- Long-range surface influences on chemical reaction rates are apparently operative in the formaldehyde clock reaction.

It might be added that the demonstration of surface effects in the formaldehyde clock reaction is an original discovery which will be reported in the open literature.

Another interesting phenomena in the foam demonstration was the observation that the fluids always stayed at the "bottom" of the tubes (toward the point of attachment to the working surface). Post-flight consideration of this phenomenon, leads to the conclusion that the shape of the tube is responsible. If a liquid is contained in a tube whose diameter varies, the liquid will position itself so as to minimize surface energy. The liquid will move from regions of large diameter to regions of small diameter (Ref. 1).

A preliminary report on the Chemical Foams experiments has been presented previously (Ref. 2).

The Liquid Spreading demonstration was expected to yield data on the wetting and spreading action of fluids on a solid surface in the space environment. When a liquid is brought in contact with a solid surface, it tends to spread over the entire surface provided that adhesive forces between the molecules of the liquid and solid are greater than the cohesive forces between the liquid molecules. In one-g, however, this wetting action generally takes place in a horizontal or downward direction; gravity tends to "pull" on a fluid mass so that the liquid will not distribute itself evenly over an object. In the space environment, without the gravity influence, a wetting fluid will spread over a surface in a manner dictated only by the properties of the fluid and solid and by the geometry of the solid body. This demonstration was designed to easily and visibly show this phenomenon.

The Liquid Spreading demonstration shows the rate of spreading of oil on Lexan and (fortuitously) on a metal surface. Additionally, the behavior of water on an oil film is demonstrated. Ground based spreading rates have been measured along with contact angles and surface tension values for the flight fluids. The experimental data fell within the bounds of existing theories, but the low-g data were not precise enough to allow a decision as to which theory was the most representative of spreading in the absence of gravity. Further experiments under more controlled conditions are indicated.

The Chemical Foams and the Liquid Spreading demonstrations are discussed in two separate subsequent sections.

REFERENCES FOR INTRODUCTION

1. Grodzka, P. G., and B. Facemire, "Three Model Space Experiments on Chemical Reactions," Paper presented at the 1976 NASA Colloquium on Bioprocessing in Space, Houston, Texas, March 10-12, 1976, Proceedings issued as NASA TMX-58191, January 1977.
2. Petrash, D. A., and E. W. Otto, "Controlling the Liquid-Vapor Interface Under Weightlessness," Astronautics and Aeronautics, March 1964, pp. 56-61.

CHEMICAL FOAMS

by

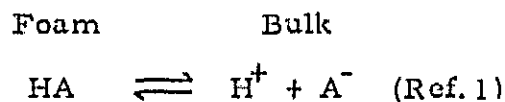
P. G. Grodzka

INTRODUCTION

The Chemical Foams demonstration consisted of two subparts; a foaming demonstration and a formaldehyde clock reaction. These are discussed separately in the next two subsections. Experimental apparatus, solution compositions, and procedures are given in the Appendix to this section.

FOAMING DEMONSTRATION

In this part of the demonstration, aqueous solutions of bromthymol blue indicator were shaken. Two different ratios of air to liquid were tried. When such solutions are shaken to form foams, the foamed portions are pink in contrast to the golden color of the unfoamed parts. The pink color is due to the following chemical reaction:



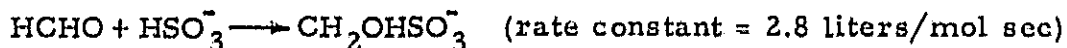
In ground tests the foam quickly dissipates due to the buoyancy of the air in earth's gravity. However, in space without the buoyancy effect, the foams were observed to be much more stable. Times for foam dissipation, however, were not determined. Also differing ratios of air to liquid were not seen to exert any appreciable effect on the foam stability in low-g. Enhanced stability was also demonstrated on Skylab with two immiscible liquids (oil and water) in Science Demonstration TV102 (Ref. 2).

THE FORMALDEHYDE CLOCK REACTION

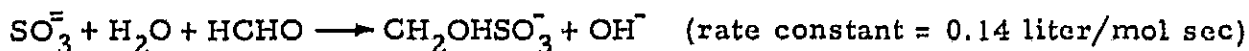
Chemical Basis: This reaction consists of mixing aqueous solutions of formaldehyde, sodium sulfite and bisulfite, and phenolphthalein. The mixed solutions react for a time interval which depends on the various concentrations of formaldehyde and sulfite/bisulfite species. After the reaction is complete, the reacted solution suddenly turns a bright red.

The chemical changes consists of the simultaneous occurrence of three homogeneous reactions proceeding at different rates. These three reactions are as follows (Ref. 3).

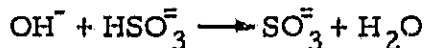
1. The addition of bisulfite ion to formaldehyde;



2. The reaction of sulfite ion and water with formaldehyde yielding OH:



3. The instantaneous reaction of OH⁻ with bisulfite ion:



Thus, excess OH⁻ becomes available for reaction with the indicator, phenolphthalein, only after the bisulfite ion has been completely used.

Flight Results and Further Ground Tests: As mentioned previously, reaction times obtained in ground and in the low-g tests were insufficiently precise to allow a decision as to whether or not the reaction occurred more rapidly in space than on the ground. Further details are given in the Appendix.

The significant aspects of the flight and ground results are summarized in the following statements.

Flight Tests:

1. Gas/liquid dispersions were produced in the low-g cases. The dispersions positioned themselves much as they would on the ground, i.e., Fig. 1.

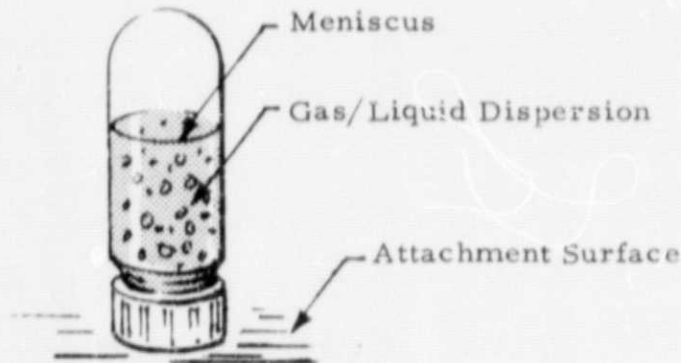


Fig. 1 - Schematic of Positioning of Gas/Liquid Dispersions in Low-g

2. The reaction initiated as evidenced by the appearance of a dark red color in the liquid meniscus and not in the gas/liquid dispersion as expected.
3. A lighter pink color developed simultaneously throughout the solution.
4. The dark red color that first developed at the liquid meniscus spread into the rest of the solution in a plane generally parallel to the gas/liquid surface. Some intensification of the homogenous pink color, however, also occurred simultaneously.

Ground Tests:

1. At room temperature in polycarbonate (Lexan) test tubes, the color change is frequently first observed in the small drops that cling to the sides of the tubes. Red spots often also occur in the solution simultaneously. A color change originating at a point in the solution and then rapidly spreading out as a wave throughout the rest of the solution will also occur, either by itself or in conjunction with the drop and spot phenomena.

2. In Pyrex test tubes at room temperature, spreading waves of color are also observed. The liquid film clinging to the glass wall, however, changes color last.
3. In foamed polystyrene containers, the color change first appears as spots, apparently originating at solid/liquid interface sites (Fig. 2) and then spreads at varying speeds depending on liquid mass and shape throughout the remainder of the liquid.
4. In a container containing small (50 to 120 mesh) glass beads, color spots, again apparently originating at the solid/liquid interface sites, are also observed.
5. The color change is almost always first observed in liquid filling cracks or crevices.
6. If a small glass capillary is inserted into the bulk liquid, the color change starts at the meniscus and in the bulk liquid about simultaneously. However, the spread of the color from the meniscus or from the bulk through the liquid in the capillary is considerably delayed.
7. If varying size drops of reacting solution are placed on Lexan or on other nonwetting surfaces at room temperature, the color change sequence most frequently seen is largest drops first, intermediate drops next, and smallest drop last. It is interesting to note, however, that this order is not consistently noted with drops that cling to the walls of Lexan tubes. Innumerable further tests established that this apparent inconsistency is caused when drops formed on the walls of Lexan tubes were formed by a rapid, sundering of shaken liquid. Also if a mixed solution is divided unequally between two like small beakers, the color change occurs most frequently in the beaker containing the most solution.
8. The temperature of reacting mixtures rises about $1\frac{1}{2}$ C during the course of the reaction. It is interesting to note that drops of about equal size when placed on different surfaces change color at different times (1 to 2 sec difference). The fastest change usually occurring on metal or glass, substances of relatively high thermal conductivity. Reaction heat, therefore, does not appear to be a significant variable in tests comparing the effects of drop size and shape.
9. Motion picture frames of the color change show that apparently at least two chemical species are reacting simultaneously, one species causing a light pink color to appear a short time before a definite red color is observed. The species causing the darker color is apparently more inhomogeneously distributed in solution at room temperature than the species causing the pink color (Fig. 3). Motion pictures also indicate that internal shear as the result of fluid flow is not the cause of the observed internal effects.
10. The surface tension of a newly diluted formaldehyde solution (i.e., 1 ml of 3.3% formaldehyde diluted to 8 ml) is approximately 58

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Fig. 2 - Color Development as Spots on Styrofoam Surfaces

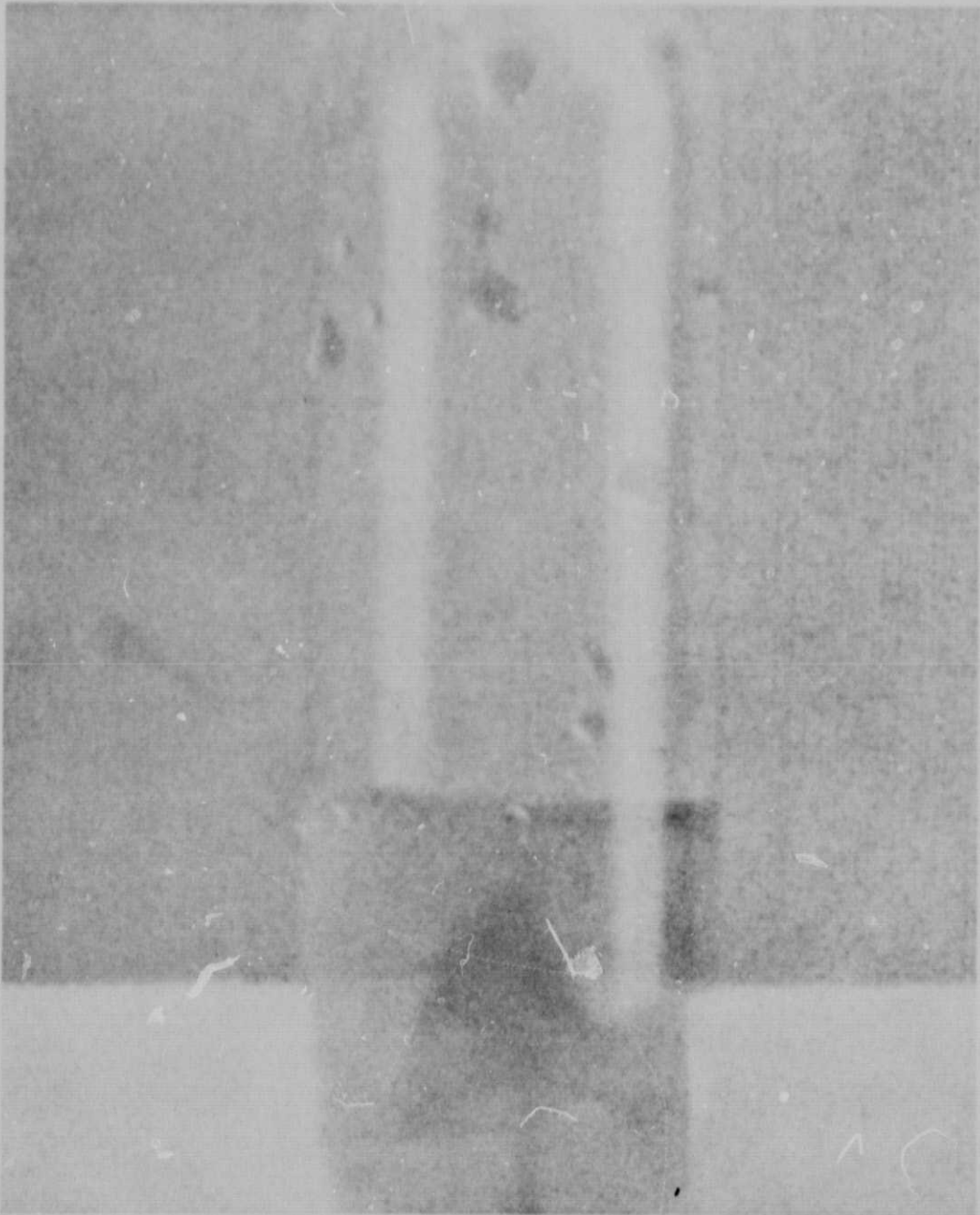


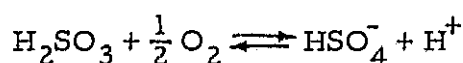
Fig. 3 - Inhomogeneous Appearance of Dark Red Color

dynes/cm initially and gradually increases to approximately 67 dynes/cm in approximately 25 min at room temperature, as shown in Fig. 4.

11. If stock formaldehyde is diluted at room temperature approximately 25 min before conducting the reaction, the color change is gradual. The solution first turns pink homogeneously, and the color gradually deepens. Drops of reacting prediluted solution on nonwetting surfaces, however, still change most frequently in the order largest first to smallest last.
12. If all the solutions are chilled in the refrigerator for a few hours before conducting the reaction, a slight foam, formed when the reactants are mixed by shaking, persists during the reaction. The color change (in Lexan tubes) in such a case proceeds as follows: drops on the wall first, foam/wall contact area next, and bulk liquid last. The spread of color throughout the bulk liquid, however, proceeds spectacularly in two complex waves, a pink wave being followed by a red wave. Less severe cooling, to approximately 16 C, also favors the wall drops changing color first. Subsequent color changes, however, are much more rapid.
13. If platinum or stainless steel electrodes, connected through a variable resistance to a 1.5 V battery, are inserted into a reacting solution, a red color appears almost immediately at the negative (cathode) electrode at voltages higher than about 0.2 V. Some "memory" behavior, however, is encountered, i.e., the first time the test is tried, about 0.8 V may be required to observe the effect. Subsequent tests, however, show the effect at voltages as low as about 0.2 V. No color appears at the electrodes if formaldehyde is omitted from the solution; i.e., the color change is not caused by electrolysis of water under the conditions used in these tests. Preferential initiation at the negative electrode, however, is not observed if the formaldehyde solution is not freshly diluted.

Discussion: Before an explanation of the preceding results is attempted, it may be well to indicate why factors such as oxygen absorption, liquid or formaldehyde evaporation effects, and thermal conductivity effects are ruled insignificant in the observed phenomena.

Absorption of oxygen from the air cannot have been a cause of the observed phenomena. Oxygen can interact with sulfite or bisulfite species through the reaction



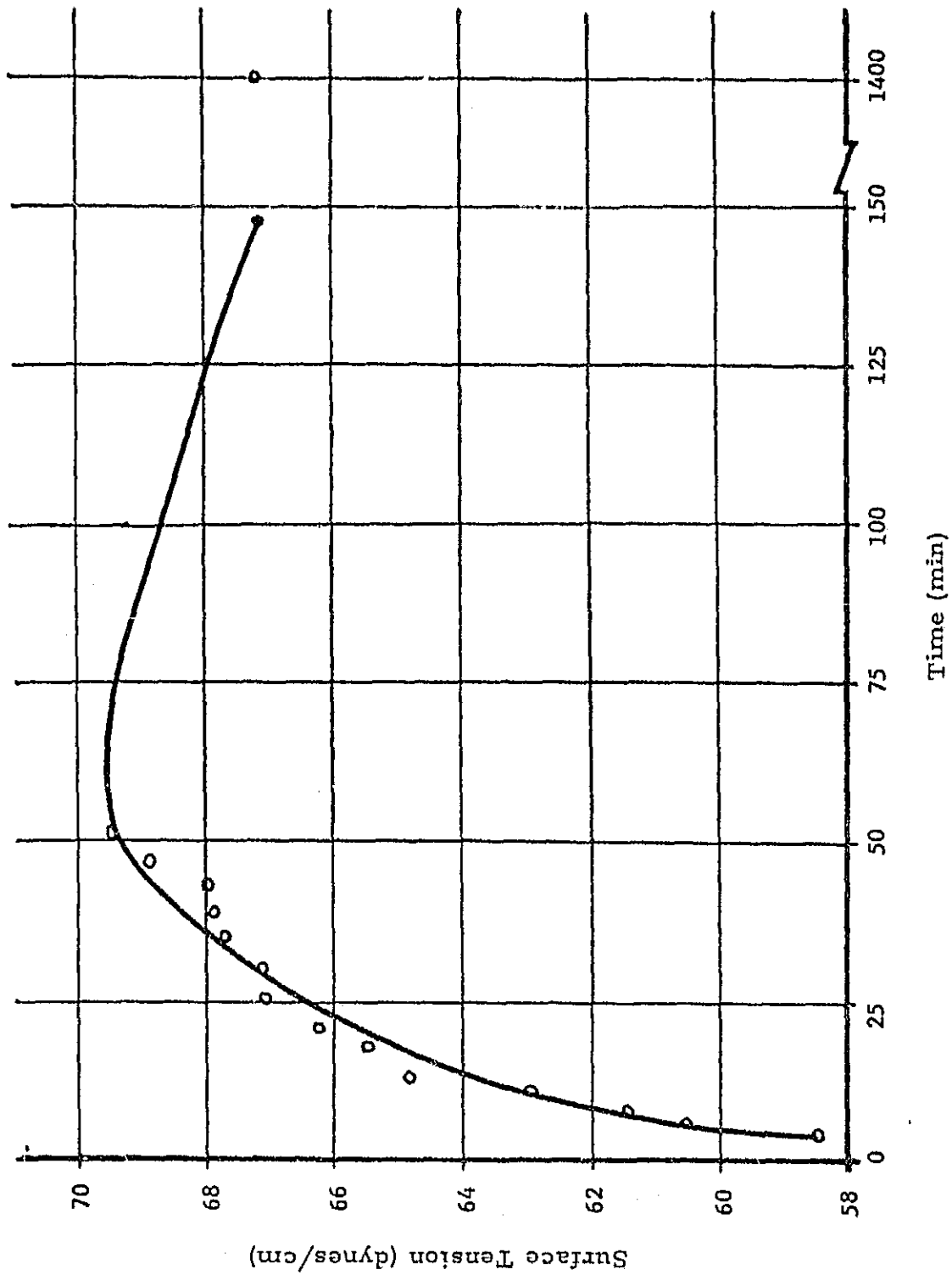


Fig. 4 - Variation of Surface Tension with Time ($T = 0$ is Time of Dilution) for 1 ml 3% Formaldehyde Added to 8 ml Distilled Water (Accuracy of these readings is $\pm 5\%$.)

The reaction is slow in the absence of catalysts. Even if it had occurred to any appreciable extent at the liquid-air interface in these tests, the effect would be to make the interfaces more acid. The onset of red color would thus be expected to be considerably delayed at liquid-air interfaces, not accelerated as is actually observed. To verify this conclusion, a test was conducted which included a nitrogen purge in a closed tube prior to the performance of the reaction. The reaction was observed to proceed in all respects the same as it does when no purging is employed. The possibility that sulfite depletion is the cause of the color appearing first in drops clinging to wall tubes can also be discounted. If this were so then small drops deposited on surfaces by means of an eye dropper should consistently change color faster than larger drops. This usually is not the case. Also, the fact that the color change could be made to occur faster in smaller drops than in larger ones by a rapid sundering of the liquid masses also reinforces the logic that oxygen absorption is not a significant variable. Finally, the fact that identical beakers containing different amounts of the same reacting mixture change at different times would tend to rule out different oxygen absorption or evaporative rates because of different surface/volume ratios. Similar reasoning can be applied to rule out the factors of evaporative and thermal conductivity cooling.

A number of the preceding data can be interpreted on the basis of formaldehyde species equilibria in solution. It is well known that aqueous solutions of formaldehyde contain a number of distinct, polymeric chemical species which are in equilibrium (Ref. 4). The display of different color development behavior and increase of surface tension depending on length of time since dilution and temperature are undoubtedly caused by species equilibria shifts. The often observed development of color at certain sites and at negative electrodes provides a vivid demonstration of chemical nucleation.

The effects of drop size, shape, and solid surface material indicate a long-ranged surface influence on chemical reaction rates. Long-range orientation has been previously discussed as a cause for some unusual

observations in film crystal growth and in the freezing of water in capillaries (Refs. 5 and 6). In the freezing of water in capillaries it was found that the freezing point of water in capillaries appears to be a function of the greatest distance any part of the specimen can be from the nearest interface. The freezing behavior of water suggests that the delayed reaction in capillaries and the other surface and size effects observed in the present study arises from a similar cause. Of course, when dealing with water in capillaries the danger is run of contamination. It is difficult to believe, however, that significant contamination could have taken place in the present study in the few seconds it took to run the reaction.

The fact that in the bulk liquid the reaction generally prefers to start at menisci rather than at small bubbles in the liquid again indicates some influence of solid surface. The observation that the reaction started first in the foam portion of a chilled sample indicates that a future low-g repeat test should be chilled in order to obtain a true foam.

Conclusions: The Chemical Foams demonstration showed that aqueous foams and liquid/gas dispersions are much more stable in low-g than in one-g. Certain chemical reactions, therefore, that are not possible on the ground should be possible in low-g foams or dispersions. Furthermore, the formaldehyde clock reaction demonstrates chemical nucleation and also behavior indicative of long-range surface influences on chemical reaction rates. As far as is known, the present report of long-ranged surface influences on chemical reaction rates is the only known report of such behavior.

The present work illustrates the serendipity effect provided by the motivation of developing a low-g experiment. Indications of the various unusual phenomena observed in the present study in the formaldehyde clock reaction were obtained by a previous worker (Ref. 7). But lacking the motivation of exploring these more fully, the phenomena were not pursued further. Thus, although the present study failed to demonstrate that chemical reactions occur more rapidly in low-g foams than on the ground, a couple of other unique phenomena were demonstrated.

Possible Applications: The implications of the space experiments with regard to possible applications fall into two areas. One area is concerned with directions for basic research on biophysical processes and the other with directions for processing applications in low-g. In the area of basic research, the formaldehyde clock reaction would appear to have relevancy to phenomena such as the clotting of blood and the formation of cataracts. For example, the following description of how a lobster's blood clots on a glass slide sounds much like a description of how a formaldehyde clock reaction occurs in a plastic tube (Ref. 8):

"A wave of changes must start at the interface between the glass and blood, and progress through the latter, involving these sensitive corpuscles in its path. . . . The two impressive features of this phenomenon are: (1) that a chemical change of catastrophic character can be started at an interface, and (2) that the change can be propagated apparently indefinitely through one of the phases."

A description of the processes involved in the formation of cataracts (Ref. 9) also sounds as if processes similar to those found in the formaldehyde clock reaction are involved. For example, the formation of regions of cortical opacities within a cataractous lens can be compared to the formation of red spots within the solution in the formaldehyde clock reaction because an opacity is caused by an abrupt or irregular change in protein concentration. Also interesting is the fact that internal structure effects appear to play a role both in the formaldehyde clock reaction and in the formation of cataracts. Also solution size or volume effects are notable in both phenomena. The hints that the two phenomena may be related are tantalizing and a number of experiments immediately suggest themselves.

In the area of processing applications, the demonstration of a formaldehyde clock reaction and an equilibrium shift reaction in low-g indicates that low-g forms can be unique environments for conducting biochemical reactions. Also the demonstration of a longer lasting foam in low-g indicates foam separation processes that cannot be done on earth because of the long times required for adsorption, i.e., the foam on earth does not last long enough for adsorption to occur.

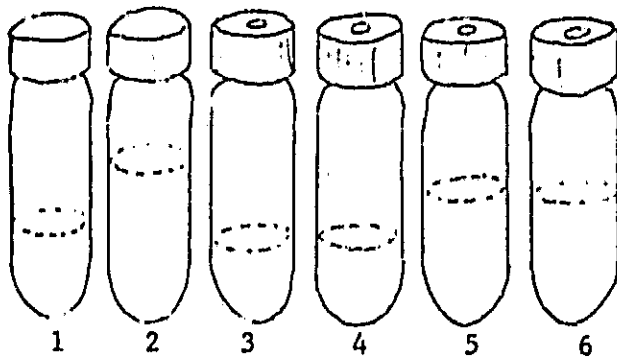
AppendixEXPERIMENTAL APPARATUS, SOLUTION COMPOSITIONS,
AND PROCEDURES FOR THE APOLLO-SOYUZ
SCIENCE DEMONSTRATION

"CHEMICAL FOAMS"

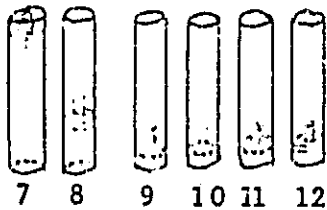
The apparatus utilized in the low-g tests is shown in Fig. A-1. The thymol blue solutions consisted of the following: 10.7 mg thymol blue (Baker), 10 ml ethyl alcohol (Baker), and 0.2 cc concentrated hydrochloric acid (Baker), all diluted to 500 ml with distilled water. Tube 1 was one-third full and Tube 2, two-thirds full. For the formaldehyde clock reaction the solution compositions were as follows: (1) 3.3% formaldehyde (9.0 ml 37.7% reagent grade formaldehyde diluted to 100 ml with distilled water); (2) 1 gm phenolphthalein dissolved in 250 ml ethanol and diluted to 500 ml with water; and (3) 10.0 gm sodium metabisulfite ($\text{Na}_2\text{S}_2\text{O}_5$) and 1.5 gm of sodium sulfite (Na_2SO_3), reagent or certified grades diluted to 100 ml with distilled water. Tubes 3, 4, 5 and 6 contained 8, 8, 18 and 16 ml, respectively, of distilled water. The procedure consisted of the Astronaut adding from preloaded syringes the following amounts of formaldehyde solution, phenolphthalein, and sulfite/bisulfite solutions to the various tubes.

Tube	Water (ml)	Formaldehyde (ml)	Phenolphthalein (ml)	Sulfite/ Bisulfite (ml)
3	8.0	1.0	0.5	0.5
4	8.0	1.0	0.5	0.5
5	18.0	1.0	0.5	0.5
6	16.0	2.0	1.0	1.0

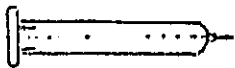
Each test was, of course, done separately. The tubes were shaken vigorously after sulfite/bisulfite addition. The temperature of the apparatus as determined by colors on a liquid crystal strip at the time of experiment performance was 24 C. Ground control tests with an apparatus that was



30 ml Lexan Tubes with Caps



Preloaded Ampules



Syringe Body (Six)

CONTENTS:

1. Thymol Blue Solution
2. Thymol Blue Solution
3. Distilled H₂O (8ml)
4. Distilled H₂O (8ml)
5. Distilled H₂O (18ml)
6. Distilled H₂O (16ml)
7. 3% Formaldehyde
8. Phenolphthalein Indicator
9. Sulfite/Bisulfite Sol'n
10. Sulfite/Bisulfite Sol'n
11. Sulfite/Bisulfite Sol'n
12. Sulfite/Bisulfite Sol'n

Fig. A-1 - Schematic of Hardware for ASTP Science Demonstration, Chemical Reaction in Foams

a duplicate of the flight apparatus gave the following times at 24 C (from moment of sulfite/bisulfite addition to observation of red color appearance):

Tube	Ground Time (sec)	Flight Time (sec)
3	16.0	—
4	25.0	—
5	27.5	—
6	21.5	22.4 or 33.6

Only one flight time was obtained because of various operational and camera problems. Even this time is in doubt because of operational problems and because the frame speed on the camera is uncertain. Thus, no conclusion can be drawn at this time as to whether or not the flight tests differed significantly from the ground tests.

In anticipation that timing data from one of the flight tests would be meaningful, a series of tests were run varying the amounts of 3% formaldehyde and water added. The results of this test are shown in Fig. A-2. All quantities were held constant except for the amount of formaldehyde in Fig. A-2. In Fig. A-3 all amounts were held constant except water. Since in flight the formaldehyde syringe backfilled with water, these are the two reactants which were changed in flight. It can be seen that a change in amount of formaldehyde has a much greater influence on the reaction time than does the volume of water. After viewing the flight film it was seen that an accurate estimate of the amount of formaldehyde actually added was impossible.

Motion pictures of the reaction were made to determine the characteristics of the reaction on earth.

Contact angle (on Lexan) measurements of each reactant, the final solution, and the thymol blue solution have been made to aid in the explanation of the phenomena of the fluids' position in the tubes. The contact angles were measured from photographs and are shown in Table A-1. Surface tension

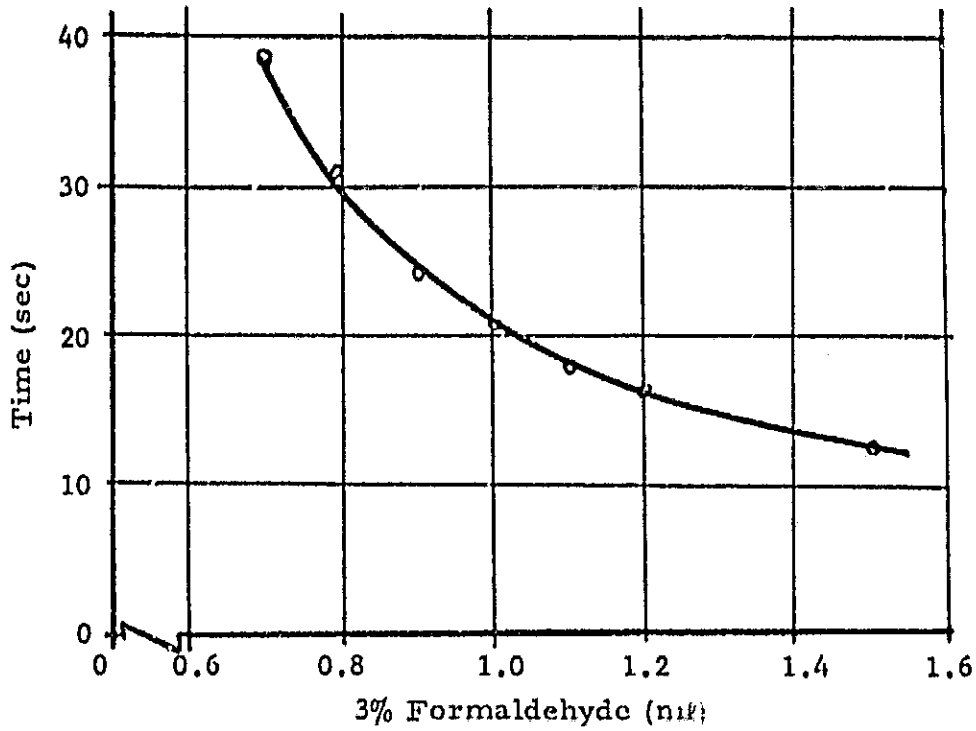


Fig. A-2 - Variation of Reaction Time with Changing Formaldehyde Additions

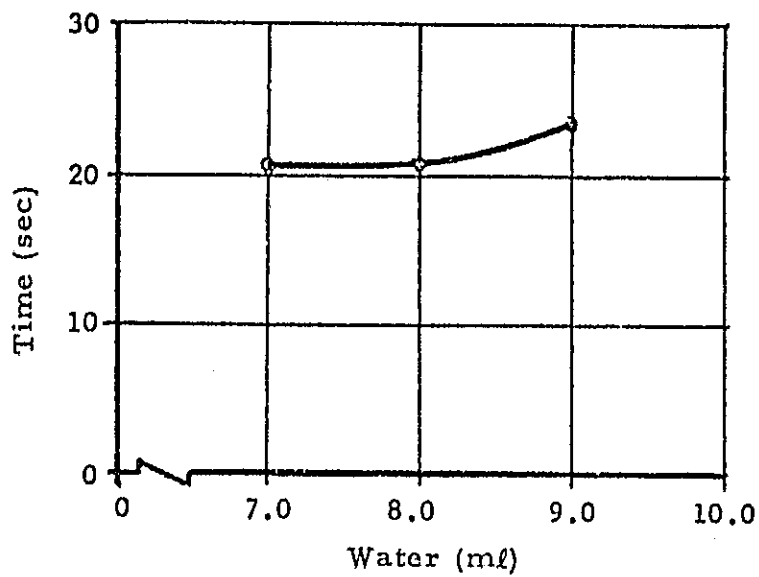


Fig. A-3 - Variation of Reaction Time with Changing Water Additions

Table A-1
CONTACT ANGLES

Material	Contact Angle (mean)	Number of Readings	Standard Deviation
Distilled H ₂ O	85.0	20	6.5
3% Formaldehyde	78.3	40	4.9
Phenolphthalein Solution	36.3	7	3.4
Sulfite/Bisulfite Solution	75.5	42	5.2
Reaction Mixture	68.8	25	4.6
Thymol Blue Solution	78.1	39	4.4

measurements are being made using a Fisher surface tensiometer (Model 20). Table A-2 shows the values for distilled H₂O, thymol blue solution, and 3% formaldehyde. It is seen that all of the solutions have contact angles less than 90 deg and hence form a concave meniscus with Lexan surfaces. The surface tension of 3% formaldehyde added to water (0.4% formaldehyde) variation with time is shown in Fig. 4 of the main text.

Table A-2
SURFACE TENSION VALUES

Material	Surface Tension
Distilled H ₂ O	69.3
Thymol Blue Solution	59.2
3% Formaldehyde	63.5

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LIQUID SPREADING
ASTP SCIENCE DEMONSTRATION

by
S. V. Bourgeois

LIQUID SPREADING ASTP SCIENCE DEMONSTRATION

Introduction

Wetting and spreading phenomena are significant in a wide variety of important processes. Some examples are adhesion casting, removal of dirt by detergent solutions, dropwise condensation, application of pesticides, and displacement of one fluid by another in soils, underground oil reservoirs and other porous media. Adhesion casting is a candidate for space processing (manufacturing conducted in the microgravity environment).

Theoretical correlations of liquid spreading over solid surfaces in the absence of gravity are given in Refs. 1 through 9. A major goal of this study is to compare these theoretical predictions to the actual spreading rates obtained in the ASTP science demonstration "Liquid Spreading". The demonstration represented one of the very first opportunities to ever observe spreading in the true absence ($10^{-4} g_{\text{Earth}}$) of gravity. Large liquid drops spreading on Earth are always initially dominated by gravity forces. Later stages of spreading are controlled by surface tension forces and/or inertia forces (Refs. 3 and 4). Thus, the ASTP data, in the form of movie film, represents the first, and only, data taken in the absence of gravity in order to observe the spreading of large liquid drops wherein surface energy forces are the controlling force.

System Description

The situation under study is shown in Fig. 1. The spreading rate is determined from the change of droplet contact radius, r , versus time, t . Spreading rate, in the absence of gravity, is theoretically controlled by surface energy properties (equilibrium contact angle, θ_{∞} , and droplet surface tension, $\sigma = \sigma_{LV}$) and hydrodynamic physical properties (viscosity, μ , and density, ρ).

Theoretical Considerations

A simple analysis of the forces causing a drop to spread can be performed to determine the relative influence of gravity and surface tension forces as a function of droplet size. For a droplet of initial diameter, D , the ratio of spreading forces, ψ , can be expressed as follows:

$$\begin{aligned}\psi &= \frac{\text{Surface Tension Force}}{\text{Gravity Force}} = \frac{\pi D \sigma \cos \theta_{\infty}}{\frac{\pi}{4} \rho g D^3} \\ &= \frac{4 \sigma \cos \theta_{\infty}}{\rho g D^2}\end{aligned}\quad (1)$$

where g represents gravitational acceleration (980 cm/sec^2 on Earth). Gravity will be negligible if $\psi \gg 1$. Utilizing Eq.(1) and setting $\psi \geq 100$ as the criterion for surface tension forces to dominate, drops of silicone oil smaller than 0.03 cm in diameter must be used for ground tests in which gravity forces are negligible. It is very difficult to measure spreading rates on droplets this small. Thus, the microgravity environment of ASTP ($g \leq 10^{-1} \text{ cm/sec}^2$), provided a rare opportunity to study centimeter-size drops spreading in the absence of gravity influence.

The above procedure was utilized to assess the spreading of other classes of liquids (crude oil, water, liquid metals). Again the conclusion is reached that drops smaller than 0.05 cm must be used for ground tests. Thus microgravity is essential for the study of surface-tension-dominated spreading for most liquids.

Russian analyses indicate the following relationships for spreading (Refs. 3 and 4):

$$r = A t^{1/4} \quad \begin{array}{l} \text{Surface energy driving force and} \\ \text{liquid viscosity resisting force} \end{array} \quad (2)$$

$$r = A t^{1/8} \quad \text{Surface energy driving force and liquid viscosity affected by solid substrate} \quad (3)$$

$$r = A t^{1/2} \quad \text{Surface energy affected by oxide layers} \quad (4)$$

where the coefficient A is determined by the physical properties of the liquid and the shape of the spreading drop. For hemispherical shapes, A is defined as (Ref. 4):

$$A = \left(\frac{6}{\pi} m \sum \right)^{1/4}$$

$$\sum = \frac{K \sigma_{SV} - (\sigma_{LV} + \sigma_{SL})}{\rho \mu}$$

where m is the mass of the liquid drop and K is the solid surface's roughness factor. The factor, K, describes roughness on a microscale and does not account for scratches, grooves, etc. The numerator of the term for $\sum$ is directly proportional to the equilibrium contact angle, θ_{∞} .

Strella's analysis of liquid spreading (Refs. 1 and 2) yields the following relationship between drop radius, r, and time, t,

$$r = R_o \sin \phi \left(\frac{4}{2 + 3 \cos \phi - \cos^3 \phi} \right)^{1/3} \quad (5)$$

$$t = \frac{\sqrt[3]{4} R_o \mu}{\sigma} \left(1 + \cos \phi_{\infty} \right)^{-2/3} \left(2 - \cos \phi_{\infty} \right)^{-1/3} B \quad (6)$$

$$B = \frac{1}{2} \ln C_1 + \sqrt{3} \tan^{-1} C_2 \quad (7)$$

$$C_1 = \left(\frac{1 + h + h^2}{1 + h_o + h_o^2} \right) \left(\frac{1 - h_o}{1 - h} \right) \quad (8)$$

$$C_2 = \frac{\sqrt{3} (h_o - h)}{2 + h_o + h + 2 h_o h} \quad (9)$$

$$h = \left[\left(\frac{2 - \cos \phi}{2 - \cos \phi_\infty} \right) \left(\frac{1 + \cos \phi_\infty}{1 + \cos \phi} \right) \right]^{1/3} \quad (10)$$

$$h_o = \left[\frac{1 + \cos \phi_\infty}{2(2 - \cos \phi_\infty)} \right]^{1/3} \quad (11)$$

$$\theta = \pi - \phi; \theta_\infty = \pi - \phi_\infty \quad (12)$$

Strella's analysis assumes that spreading is driven by surface energy forces and resisted by liquid viscosity forces.

Varma et al. (Ref. 8) have recently developed a spreading (liquid-on-solid) model in which surface tension alone is the driving force, while the resisting force consists of liquid viscosity. Their model neglects gravity, inertia and capillary forces (from scratches and microporosity). They utilize Young's equation (a line force) for the surface energy forces, assume the liquid to be incompressible and restrict the equilibrium contact angle to values less than or equal to $\pi/4$. The resulting expression for drop contact radius as a function of spreading time is an implicit relationship, however, it yields the following for silicone oil spreading on Lexan:

$$r = A t^{1/5}$$

for $\tau > 1$, where:

$$\tau = \sqrt[3]{4} \sigma t / R_o \mu$$

The latest analysis and model of liquid-solid spreading is due to Lopez, Miller and Ruckenstein (Ref. 11). Their analysis is applicable when: (1) the

drop has spread over an area much greater than it initially covered, and (2) the spreading rate is controlled by the balance between viscous resistance to flow and a single dominant driving factor, either gravity or surface forces near the drop's periphery. The surface forces are not modeled by a simple line force (Young's equation). Instead the intermolecular forces throughout the thin liquid layer near the droplet edge are considered (Ref. 9). Furthermore, surface diffusion is neglected and only cases where the liquid spreads spontaneously ($\theta_\infty = 0$) are considered.

For the case of an axisymmetric drop, Lopez et al. (Ref. 11) predict the following relationships for the growth of droplet contact radius, r , as a function of spreading time, t :

$$r = A_1 t^{1/2} \quad \text{Surface tension}$$

$$r = A_2 t^{1/8} \quad \text{Gravity force}$$

More rigorous theories exist for liquid-liquid spreading (e.g., oil on water) and yield the following rates (Refs. 6 and 7):

<u>Driving Forces</u>	<u>Spreading Rate</u>
Inertia	$r = A t^{1/2}$
Gravity	$r = A t^{1/2}$
Surface Tension	$r = A t^{3/4}$

The preceding theories each indicate that circular spreading of drops can be described by the general relation:

$$r = A t^n$$

where n varies between $1/8$ and $3/4$.^{*} The parameter A is dependent upon physical properties of the liquid, but is a constant for a given, specified system; i.e., silicone oil spreading on Lexan being driven by surface tension. The variation of the exponent, n , as a function of driving and resisting forces of spreading is summarized in Exhibit 1.

It is evident from Exhibit 1 that the theory for spreading on solids is not as complete as that for spreading on liquids. Thermodynamic aspects of wetting and contact angles are generally well understood, but kinetic aspects are not (Ref. 11) as evidenced by the range of values for time exponent, n , in the first row of Exhibit 1. Note also that the effect of inertia forces is not available for the case of spreading on solids. If one assumes that spreading behavior on solids and on liquids are similar, then the following conjecture^{**} may hold for exponent of time, n :

$$\frac{n \left| \text{Liq-Liq-Inertia} \right.}{n \left| \text{Liq-Liq-Surface Tension} \right.} = \frac{n \left| \text{Liq-Solid-Inertia} \right.}{n \left| \text{Liq-Solid-Surface Tension} \right.}$$

therefore,

$$\frac{1/2}{3/4} = \frac{n \left| \text{Liq-Solid-Inertia} \right.}{1/4}$$

and,

$$n \left| \text{Liq-Solid-Inertia} \right. = 1/6$$

^{*}Strella's analysis results in $n = 1/2$ for initial spreading and $n = 1/4$ for later stages of spreading.

^{**}Assuming that Refs. 1 through 4 are the correct theories.

Thus, one can approximate inertia-dominated spreading on solids by:

$$r = At^{1/6} \quad (16)$$

In deriving the expressions for spreading embodied in Eqs. (2) through (16), various assumptions and simplifications were utilized (Refs. 1 through 4, 6 and 7). A summary of these simplifications are given in Exhibit 2 for the case of spreading over solids. None of these assumptions should invalidate the general time dependency relationship for spreading; i.e., the slope of drop-let contact radius versus time on a log-log plot should be equal to the value of the exponent, n , given in Eqs. (2) through (16) and in Exhibit 1.

Data Analysis

Equations (5) and (6) were utilized to plot r versus t on log-log paper for the liquid and solid surfaces utilized in the ASTP demonstration (DC 200 silicone oil and Lexan). The physical properties for this oil are as follows:

$$\begin{aligned} \sigma &= 19.74 \text{ erg (gr/sec}^2\text{)} \\ \mu &= 0.0934 \text{ gr/cm-sec} \\ \rho &= 0.93 \text{ gr/cm}^3 \\ \theta_{\infty} &\approx 1.7 \text{ deg} \end{aligned}$$

The ASTP spreading data are to be compared with these theoretical curves. In addition to displaying and comparing data in the above manner, the exponential parameter, n , is calculated for every ASTP data point and an overall average is determined using all these data points. This average value will be compared to theoretical values given in Exhibit 1.

The spreading rate curve is shown in Fig. 2 for initial drop radii of 0.11, 0.14, 0.155, 0.215, 0.31, 0.46 and 2.5 cm. Calculations required to solve Eqs. (4) through (11) have been computerized. The preceding radii are the initial droplet sizes observed for the ASTP demonstration. Flight data are also shown on Fig. 2. A more detailed discussion of ASTP data are given later.

The theoretical spreading rate exhibited in Fig. 2 indicates that initial spreading is proportional to $r = A t^{1/2}$, whereas final spreading follows the expected (from Russian analysis) $r = A t^{1/4}$ relationship. This proves that the Russian analysis is not valid for the initial stage of spreading; however, once the drop shape becomes hemispherical the Russian analysis agrees with that of Strella. It should be noted that the curves of r versus t for $\theta_{\infty} = 5$ deg is identical to those shown in Fig. 2 ($\theta_{\infty} = 1.7$ deg). Thus, contact angle apparently has only a small effect on spreading rate as long as $0 \text{ deg} < \theta_{\infty} < 5 \text{ deg}$.

Data obtained from ASTP consists of spreading rates (r/R_0 versus time) for five drops of silicon oil spreading on Lexan and one drop spreading on an aluminum tray.* A summary of these flight data are given in Table 1 and individual values for $(r/R_0, \tau)$ are also shown in Fig. 2.

Utilizing the data in Table 1 for the six drops, the value of the exponent n in the expression $r = A t^n$ is found to average 0.19. From the theoretical discussions presented earlier, values of $n = 0.20, 0.25$, or 0.50 should be expected for surface tension forces controlling, whereas a value of $n = 0.167$ should result in inertia forces (from the astronaut's initial deployment of the drop) are significant.

Since the ASTP-derived value of n was between the lower limit for surface forces and that of inertia dominance, we conclude that the astronaut deployed the droplets too hard and that true surface tension controlled spreading was not obtained in the ASTP demonstration.

*Details of the experiment apparatus will be given in the final report.

Droplet Impact

In addition to data on spreading drops, the ASTP demonstration also yielded data for several drops which impacted the sidewalls at relatively high velocity. The impact, and subsequent splatter (rebound) and/or spreading, of liquids on solid surfaces is of interest in the following fields of study: turbine blade cooling by impinging drops, aircraft icing, rain drop erosion, flash boilers and impingement atomizers (Ref. 12) and bouncing of drops from aerosol filters (Ref. 13).

None of the ASTP drops splattered upon impact with the walls. This is in agreement with the available theory (Refs. 12 and 13). The case studied in Ref. 12 which matched the conditions of ASTP is as follows:

$$\sqrt{g/R_0}/V = 0$$

where V represents the droplet velocity.

Conclusions

The spreading rates of liquid droplets on solids which were achieved in the ASTP science demonstration represented a unique opportunity for the long-term study of large drops spreading in the absence of gravity effects. The data on spreading rates fell within the bounds of existing theories. Available theories, however, are not in agreement on spreading rate for the case of surface forces controlling the spreading. The ASTP data, unfortunately, were not quantitative enough to select which theory is most accurate. Thus a better controlled, repeat of this spreading experiment in another microgravity experiment opportunity (drop tower, sounding rocket, or shuttle) is needed to determine which of the existing, competing models is accurate.

The microgravity environment is essential to the conduct of this experiment because of the extremely small drops required to eliminate gravity effects in terrestrial tests (≤ 0.05 cm). In addition to settling a scientific controversy,

confirmation of theory for surface controlled, liquid-on-solid spreading is required and important if such promising space manufacturing techniques, such as adhesion casting, are to be developed and proved feasible.

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NOMENCLATURE

Symbols

A	a coefficient in the spreading rate expressions, see Eqs. 2-4, 16
B	a coefficient defined in Eq. 7
C_1, C_2	coefficients defined in Eqs. 8 and 9
D	liquid droplet diameter before contacting the solid surface, cm
g	gravitational acceleration, cm/sec ²
h, h_0	coefficients defined in Eqs. 10 and 11
K	roughness factor of the solid surface
m	mass of the liquid droplet, gr
n	the exponential factor for time in the spreading rate expressions, See Eqs. 2-4, 16
r	radius of the contact area formed between the liquid droplet and the solid surface, cm
R_0	$D/2$, cm
t	elapsed time after liquid drop touches solid surface, sec

Greek

μ	bulk viscosity of the liquid, poise
ϕ	supplement of the contact angle, θ ; $\phi = \pi - \theta$
ϕ_∞	supplement of the equilibrium contact angle, $\phi_\infty = \pi - \theta_\infty$
π	3.142 radians, 180 deg
ρ	liquid density, gr/cc
σ, σ_{LV}	surface tension of the liquid-vapor interface, dyn/cm or erg/cm ²
σ_{LS}	surface energy of the liquid-solid interface, dyn/cm or erg/cm ²
σ_{SV}	surface energy of the solid-vapor interface, dyn/cm or erg/cm ²

NOMENCLATURE (Concluded)

Σ	$\left[K \sigma_{sv} - (\sigma_{LV} + \sigma_{SL}) \right] / \rho \mu$
θ	instantaneous contact angle of the liquid-vapor-solid contact lines, the angle between the tangent to the 3 phase contact line and the liquid-solid interface
θ_{∞}	equilibrium contact angle
τ	dimensionless time, $\sqrt[4]{4} \sigma t / R_o \mu$
ψ	ratio of surface tension to gravity spreading forces, $4\sigma \cos \theta / \rho g D^2$

Exhibit 1

THEORETICAL RELATIONS FOR TIME
DEPENDENCY OF LIQUID SPREADING

Liquid Spreading Over	Controlling Driving Force	$r = A t^n$ Primary Resisting Force	n	Refs.
Solid	Surface Tension	Bulk Viscosity of the Liquid	1/5 1/4 1/2	6 1, 2, 3, 4 9
Solid	Surface Tension	Viscosity of the Liquid Changes Due to Interaction of the Solid with the Spread- ing Liquid [†]	1/8	2, 4
Solid	Surface Tension	Bulk Viscosity of the Liquid and Liquid Surface is Coated with an Oxide Layer or "Scum"	1/2	4
Solid	Gravity	Bulk Viscosity of the Liquid	1/8	9
Liquid	Surface Tension	Bulk Viscosity of the Liquid	3/4	6
Liquid	Gravity	Bulk Viscosity of the Liquid	1/4	6, 7
Liquid	Inertia	Bulk Viscosity of the Liquid	1/2	6

* r = liquid drop contact radius

A = constant

t = time

$$\mu = \mu_{\text{initial}} t^{1/2}$$

for liquid titanium spreading over the surface of polycrystalline graphite due to carbonization of the titanium

Exhibit 2

SUMMARY OF ASSUMPTIONS UTILIZED IN THEORIES
FOR LIQUID DROPS SPREADING ON SOLIDS

Russians (Refs. 3, 4):

- Young's equation is valid
- θ_{∞} is approaching 0.0
- Drop's sphere shape approximated by an equal sized cone
- Vertical velocity gradient is constant over the thickness of the liquid layer
- Accelerations in the flow (inertia), gravity and capillarity are neglected

Strella (Refs. 1, 2):

- Young's equation is valid
- Cylindrical shaped drop
- Shear field approximated by that of the uniform compression of a cylindrical rod of equal volume, taking changes in the height of the horizontal rod as the measure of strain
- Inertia, gravity and capillarity forces neglected

Varma et al. (Ref. 6):

- Young's equation is valid
- $\theta_{\infty} \leq \pi/4$
- Cylindrical drop shapes
- Shear field approximated by "lubrication theory"
- Inertia, gravity and capillarity forces neglected

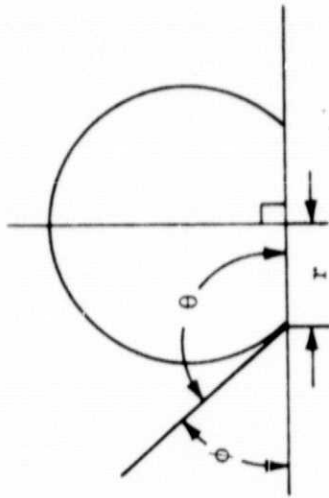
Lopez et al. (Ref. 9):

- Do not invoke line force (Young's theory)
- $\theta_{\infty} = 0$
- Cylindrical and axisymmetric drop shapes included
- Shear field approximated by "lubrication theory."
- Inertia and capillarity forces neglected.

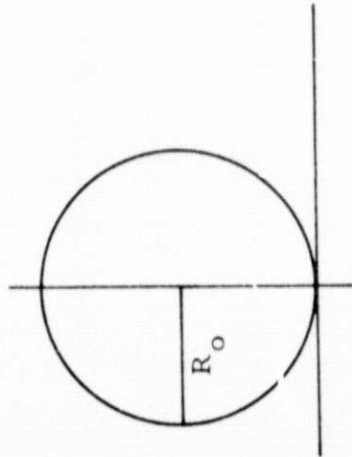
Table 1
ASTP SPREADING DATA (DC 200 SILICONE OIL SPREADING
OVER LEXAN AND ALUMINUM SURFACES)

Droplet Identification Number	Initial Radius R_o (cm)	Spreading Rate			Average Value of Time Exponent ($n = A t^n$) $n = \frac{\Delta (t^n r/R_o)}{\Delta t}$
		r/R_o	$\tau = \frac{\sqrt[3]{4} \sigma t}{R_o \mu}$	t (sec)*	
F1	0.215	2.94	0.56	0.03	0.40
		3.51	1.42	6.50	
		4.37	2.33	10.66	
		5.73	3.24	14.82	
F2	0.46	1.48	0.07	0.69	0.15
		1.90	0.52	5.09	
		2.09	0.92	9.00	
F3	0.11	7.22	1.00	2.34	0.17
		8.00	1.80	4.21	
F4	0.14	5.83	0.35	1.04	0.14
		6.48	1.75	5.21	
		6.65	3.15	9.38	
		8.17	5.95	17.72	
F5	0.31	3.33	0.30	1.98	0.11
		3.58	0.90	5.94	
		4.13	2.16	14.25	
		4.44	4.18	27.57	
F6	0.155	2.58	0.15	0.49	0.19
		3.80	1.10	3.63	

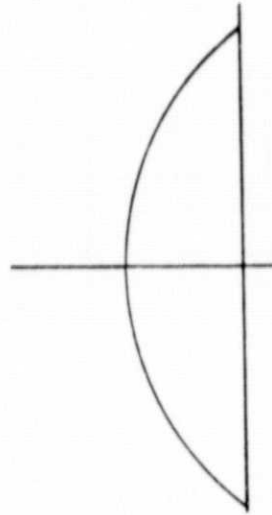
* Utilizing a film rate of 24 frames/second.



b. Intermediate Spreading Position (θ = intermediate value of contact angle, $\phi = \pi - \theta$, r = intermediate contact radius)



a. Initial Deployment of Drop onto Solid Surface ($\theta_{\text{initial}} = 180 \text{ deg}$, $r = 0$)



c. Drop Ceases Spreading ($\theta_{\infty} = 52 \text{ deg}$, $r = 6.25 \text{ cm}$ (2.46 in.))

Fig. 1 - Spreading of a Liquid Drop Over a Flat Solid Plate in the Absence of Gravity and Inertia Forces

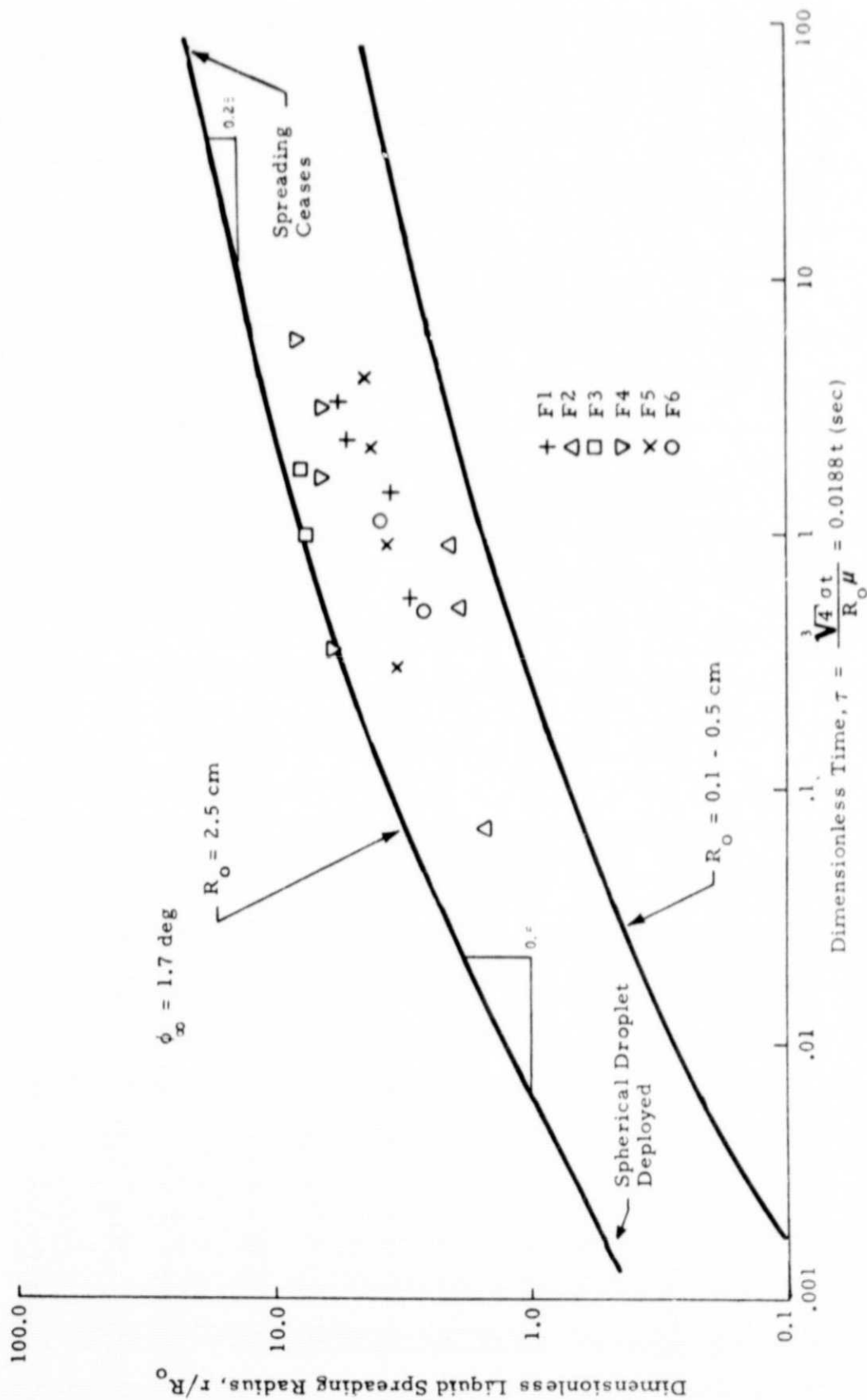


Fig. 2 - Theoretical Spreading Rate for DC 200 Silicone Oil on Lexan in the Microgravity Environment of ASTP